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Cure Monitoring Techniques Using Embedded Sensors

Roger Vodicka

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ABSTRACT

Assessing the exact state of cure of thermosetting polymer composite components using embedded sensors can allow the user to precisely control the cure cycle in order to achieve optimum cure. This is desirable in a field repair situation where a non-optimum cure cycle may have to be adopted. At present a wide variety of options are available. However, to be practical for bonded composite repair, especially field repair, it is important to assess these options against a range of criteria such as size, complexity, cure sensing ability, cost and the ability to measure other information after cure.

The techniques reviewed fall into a number of categories defined by the property which is measured. These are electrical, acoustic, optical, thermal and other/indirect properties. Of the techniques reviewed three techniques show most promise: viz. dielectrometry (electrical), spectroscopic (optical) and measurement of refractive index during cure (optical). The simplest of these techniques relies on the change in refractive index of the matrix resin during cure. A segment of cured resin is placed as a link between two lengths of a silica optic fibre. This fibre can then be placed in the uncured matrix. Light is transmitted through the fibre prior to cure but as the refractive index of the uncured resin approaches that of the cured resin the transmitted light intensity decreases until the fully cured state is achieved where no light is transmitted.

The details of this refractive index technique are highlighted and show that a cure sensing system can be simple, low in cost and effective. Therefore this technique is recommended for further investigation.

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Executive Summary

Polymer composite materials are manufactured using a combination of heat, pressure and vacuum. This transforms or 'cures' the polymer resin into a stiff, solid material through a series of complex chemical reactions. The ideal combination and timing of the cure conditions is critical to the final product. Variations in batches of material as well as material ageing can alter the optimum cure conditions. The high cost of raw materials and labour makes the rejection of parts due to incorrect cure conditions very costly. There is a need to ensure that composite parts can be consistently manufactured to a high quality standard. Techniques which can monitor the cure process and ensure optimum cure are the aim of this review. Such techniques are also particularly useful in manufacturing and applying bonded composite repairs. Repairs must be made to an exacting standard and rejection of a repair can involve a long and complicated process of repair removal.

Embedded sensors provide intimate information about the composite material as it cures. This is essential in controlling the cure process. Controlling cure conditions is often very difficult outside a laboratory. In repairs the temperature is often difficult to control due to differences in the thermal mass beneath the repair. In such situations it may be crucial to be able to evaluate the state of cure. Such sensors may in fact become a necessary part of the quality control of bonded composite repairs.

A number of techniques are currently available which fulfil the purpose of a cure monitoring sensor. Many of these may be embedded and provide detailed information on the curing composite. Furthermore, some sensors may continue to yield other information after their use during cure expires. For example this may include the monitoring of in-service flight loads or the integrity of the composite repair over the life of the aircraft.

This technical note reviews these techniques and makes recommendations on their potential. These recommendations are based on sensor size, complexity, cost, sensitivity and the ability to be used to monitor the component after manufacture. This review finds a technique based on changes in the refractive index of the resin to be most promising. Further investigation into the potential for this sensor to be used in practical field repair situations is recommended.

Contents

1. Introduction

Normal cure procedures for thermosetting composite materials follow a recipe of temperature, vacuum and pressure. Such an approach does not take account of material batch variations, material age and deviations from the recommended cure cycle in field operations. The use of a sensor which directly monitors some critical property of the cure process is required to give confidence in the manufacturing process. Such sensors may also be used in a feedback loop to drive the application of temperature, pressure and vacuum. Sensors which may be embedded in the composite provide information about the cure process during manufacture but may also be used to monitor the integrity of the component during service.

The monitoring of the curing process in epoxy resins is desirable since it allows the manufacturing process to be controlled and monitored. It is perhaps an important quality control tool, especially in situations where curing conditions are not ideal or if the matrix has undergone a cure cycle not recommended by the material manufacturer. The use of a suitable cure monitoring sensor may lead to the production of high quality parts on a consistent basis. This is important in applications such as bonded composite repairs where optimum cure cycles are difficult to achieve and are particularly critical to the final product.

The application of a suitable cure monitoring sensor to bonded patch repairs is desirable for a number of reasons:

1) Repairs are often made in a difficult environment. This includes unknown thermal mass and properties of the repair surface and the fact that the cure is not within the ideal environment of the laboratory.
2) It may be difficult or impossible to utilise traveller coupons to assess cure integrity.
3) The patch must be cured to the highest possible mechanical integrity the first time it is applied. Re-application is time consuming and may create further problems.

This technical note lists a number of cure monitoring techniques and sensors suitable for embedding into composite components and discusses their applicability in bonded composite repair. The most promising of these techniques are discussed in detail.

2. Sensor Placement

Many of the sensors mentioned in this report will give information about a localised area within the entire component. This is also a limitation of the most popular type of cure sensor, the thermocouple. In production thermocouples are placed at numerous locations over the composite tooling and part. The same approach may not be feasible for expensive or complex sensors.

It is therefore important to use sensors to monitor the most critical areas of a composite component. It is best to instrument regions which are most likely to undergo a runaway exotherm reaction or regions which are likely to receive undercure. These areas may be chosen on the basis of part thickness, thermal mass behind the curing part and other important process variables. Correct sensor placement is vital to their effectiveness.

3. Summary of Techniques

There are many techniques and sensors which have been used to measure the extent of cure in epoxy based composite materials. A complete description of all these techniques is beyond the scope of this technical note. A useful reference describing a very wide range of sensor designs is given in [1]. Sensors may measure the extent of cure directly or indirectly via some calibration or model of the process. An example of a direct measure may be determining the chemical spectroscopic make-up of the resin at any point in time. An indirect measure may be to determine temperature.

The suitability of a sensor for embedding into a composite and cure monitoring of composite structure/repairs will be judged on the following criteria.

1) It's effectiveness in assessing the state of cure

2) Ability to be embedded into a composite component

3) It's size, weight and complexity

4) Cost

5) Applicability to a wide variety of components and conditions

6) Multi-parameter sensing

The last point is important in that it is desirable to make the overall number of sensors as unobtrusive as possible. A multi-parameter sensor may be used during cure to measure extent of cure and then be used to measure another property such as temperature or strain during service. This multi-parameter sensing ability demands a great deal from a single sensor and is not possible in many cases.

The techniques currently developed for cure-monitoring may be broadly classed into five areas:

- Electrical Properties
- Acoustic Properties
- Optical Properties
- Thermal Properties
- Indirect/Other Properties

3.1 Electrical Measurements

Electrical measurements include capacitance, conductance, dielectric constant and dielectric loss tangent. Dielectrometry has created much interest in cure monitoring but has not been used extensively in production environments. Electrical techniques are subject to electrical interference in a processing environment and need to be carefully shielded when used with conductive fibres such as graphite.

3.1.1 Dielectrometry

Dielectric monitoring has been around for a long time and is popular for use in cure monitoring systems. The dielectric technique does have a number of shortcomings [2]. Degree of cure and viscosity are not easily deduced from resulting capacitance and dielectric loss tangent curves. Ciriscioli and Springer examined two resin systems in [2] using a commercially available dielectrometer. It was found that the point of lowest viscosity and the point where cure is nearly complete may be deduced readily but profiles of viscosity and degree of cure versus time were not produced accurately. Therefore this technique has applicability in basic cure monitoring but not in control of the curing process. Ketema/Programmed Composites Incorporated [3] have demonstrated the use of dielectric signature curves to control a process. The signature curve does not directly relate to a physical measure of cure but enables control of a process based on the dielectric curve of an ideally curing composite.

Dielectrometry requires the user to either embed a sensor into the composite or to place it on top of it. In either case, to use the sensor in the presence of electrically conducting fibres such as graphite the sensor must have a dielectric cavity which excludes fibres and fills with resin as the part cures. The shortcoming of placing the sensor on top of the part is that the resin must be in a very liquid state before the sensing cavity fills with resin. This negates the advantage of knowing the early changes in viscosity which are critical for correct pressure application. Sensors from Micromet Instruments Inc. are available with a thickness of about 0.15 mm and sense an area 2.5cm by 1cm. Commercially available dielectric sensing units and sensors are readily available and have found their way into some production processes.

3.2 Acoustic Methods

These techniques involves the use of both ultrasonic wave propagation techniques as well as acoustic emission. Ultrasonic measurements may be correlated with parameters such as degree of cure, porosity, viscosity, delaminations and fibre volume fraction. Acoustic emission also allows the cooling phase of a cure cycle to be monitored to determine whether thermal stresses are cracking the composite. Problems can arise when a number of these parameters are changing simultaneously as it may not be possible to distinguish between them. These techniques have not found their way into mainstream cure monitoring.

3.3 Optical Properties

3.3.1 Spectroscopic Sensors

These sensors provide information on the chemical changes which are occurring in the resin. The degree of cure may be related directly to the amount of unreacted resin. Sensors of this type tend to utilise optic fibres which need to be embedded carefully into the part during layup. Optic fibres have been used to measure strains within composites by [4]. It may be possible for the same optic fibre to be used for this purpose after it has been used as a cure sensor. The apparatus required to run these type of sensors is usually both costly and bulky.

3.3.1.1 *Infrared*

Techniques which measure the infrared spectrum of the curing resin are perhaps the most useful since actual chemical information is being gained. Such techniques can also require special fibres (eg: chalcogenide) which transmit light in the infrared region [5]. These fibres are generally expensive and only have a single use. Other types of infrared spectroscopy include Raman which has a low sensitivity and requires costly instrumentation. Recently work by Myrick et al. [6] has shown that low-cost fiber-optic Raman spectroscopy for cure-monitoring is viable using 200μm silica fibres.

3.3.1.2 *UV-Visible*

The fluorescence of chemical species in the UV/Visible region can be used for cure monitoring. Probe molecules which have a strong fluorescence in the UV/Visible region but do not take part in the curing process may be used if the signal from the neat resin itself is insufficient. The necessity of adding such probe molecules to the resin may make it difficult for use with commercial prepregs. In many cases the UV/Visible spectra derived from these probe molecules can depend on other parameters such as resin viscosity rather than actual degree of cure. Such techniques have not been widely used.

3.3.2 Refractive Index

This technique works on the fact that refractive index of the resin changes as the cure proceeds. The correlation between refractive index and extent of cure must first be established using a more direct measure of cure. A novel technique utilising this property and optic fibres appears as Section 4 below.

3.4 Thermal Properties

3.4.1 Thermocouples

Thermocouples are perhaps the simplest form of cure monitoring. They ensure that the part is up to temperature and that the cure cycle specified is in fact achieved. Their

worth as a cure sensor is very limited except for the fact that an exothermic reaction can be detected.

3.4.2 Heat Flux Sensors

This method is similar to that of a Differential Scanning Calorimeter (D.S.C) which measures heat flux versus time under a controlled temperature program. The method received attention from Perry and Lee [7]. Their system monitored the heat released during cure (the reaction exotherm) and related it to the extent of cure. D.S.C data is used widely in chemorheological models of the resin curing process. No commercially available units are on the market at present and the use of heat flux sensors is not widespread in the cure monitoring area.

3.5 Indirect Properties

3.5.1 Displacement Transducers

Displacement transducers are useful for monitoring the compaction of a part during cure. If pressure application is to be optimised this type of sensor may be essential.

3.5.2 Pressure Sensors

Sensors of this type provide information on pressure in a localised area. The sensors operate using a capacitive effect which directly correlates to pressure after appropriate signal conditioning. The pressure measured by this sensor may differ to autoclave pressure in areas where the part is contoured. This type of information is essential if accurate determination of part compaction is required. Furthermore the sensor can alert the autoclave operator of failure in the vacuum bagging material during the vacuum cycle. Dual function pressure/temperature transducers are available and can form a very functional sensor combination. Such sensors have been marketed by Interlink Electronics under the CURITE name [8].

3.6 Conclusions

Table 1 lists the techniques and their performance against the selection criteria listed in Section 3. This table shows that from all the available sensors reviewed three techniques show the most promise; dielectrometry, spectroscopic techniques and measurement of refractive index. The refractive index technique is quite new and relatively unexplored for in-field or commercial use. Excellent potential exists for such a technique to be used effectively for cure monitoring and is discussed in more detail in Section 4.

Table 1: Summary of performance of various techniques for embedding and cure monitoring of composite patches

Technique	Cure Sensing Ability	Size, Complexity	Cost	Multi-parameter sensing	Embed into composites
Dielectric	HIGH	LOW	MED	NO	YES
Acoustic	MEDIUM	LOW	MED	YES	YES
Spectroscopic	EXCELLENT	MED-HIGH	HIGH	NO	YES
Optical Refractive Index	GOOD	LOW	LOW	YES	YES
Thermocouple	LOW	LOW	LOW	NO	YES
Heat Flux	GOOD	MED	MED	NO	YES
Pressure Sensor	LOW	LOW	MED	NO	YES
Displacement Transducer	LOW	LOW	LOW	NO	NO

4. Refractive Index Cure Sensors

Work by [9, 10] has shown that changes in refractive index take place as a thermosetting polymer matrix cures. This information correlates well with other cure sensing techniques such as Differential Scanning Calorimetry (D.S.C). Refractive index sensors are available to measure this property directly and accurately but they are both expensive and may only be used once. Such sensors are available commercially from Photonetics [11]. Their METRICOR range of probes are capable of measuring pressure, temperature and refractive index. Fiberoptic refractive index probes are inherently free of electrical noise and have a resolution of about 1% over their sensing range. However the present series METRICOR refractive index sensors probes are capable of measuring refractive indices up to 1.52 which is short of the range required for cured epoxy (1.59).

4.1 Afromowitz Technique

A novel approach by Afromowitz [12] utilises a refractive index sensor which does not require calibration. The sensing setup is composed of two lengths of optic fibre joined by a short section of cured resin (see Figure 1). The entire sensing element is placed within the composite layup and a beam of light from a laser diode shines through it. The critical angle for total internal reflection changes as the refractive index of the resin changes during cure. This in turn affects the amount of light which is transmitted through the optic fibre. A signal of light transmission versus time can then be used as a

measure of extent of resin cure. As the resin reaches full cure transmission of light through the fibre ceases.

A refractive index cure sensor based on transmitted light intensity is proposed here as the most appropriate sensor based on the above selection criteria. The cost of the sensor is likely to be low since it incorporates cheap communications grade optic fibre, laser diodes and detectors. The complexity of the system is also low and it is suitable for embedding into a composite layup.

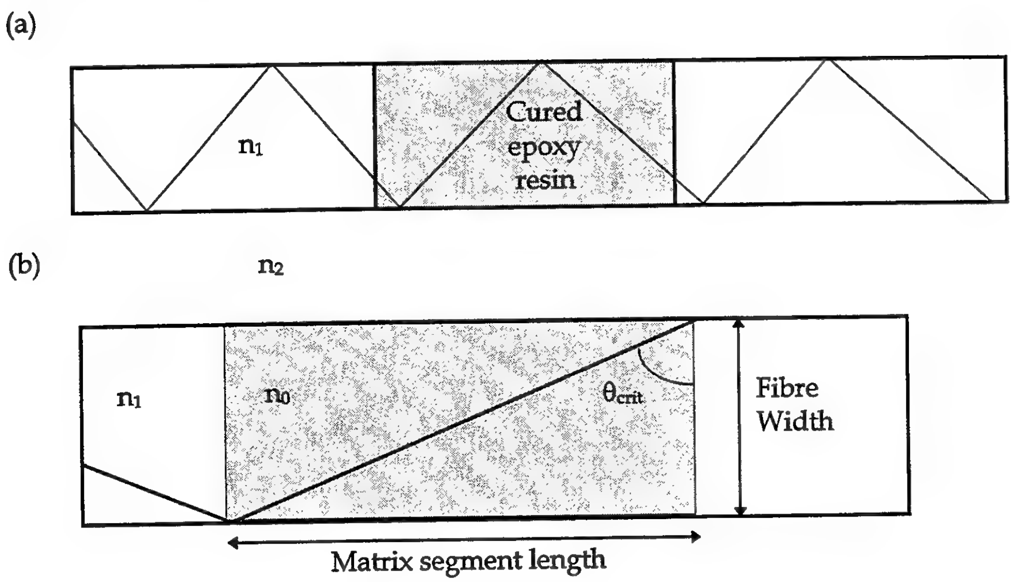

Figure 1. Schematic of optic fibre (a) and cured resin acting as a wave guide for the transmitted light (b). Refractives indices n and critical angle θ_{crit} shown.

The refractive index of the resin changes by 2 to 3% during cure so the detection of absolute refractive index may not be the most sensitive way of detecting changes. The type of sensor described by Afromowitz [12] does not require calibration providing the segment of cured polymer in the sensing element is chemically identical to the composite matrix. Although it would be possible to relate refractive index to degree of cure this may be quite a difficult task to achieve in practice. This would mean that to fully correlate the sensor signal to absolute degree of cure a chemo-rheological model of the resin would need to be formed first. This would be time consuming and increase the overall complexity.

4.2 Sensor Design and function

Total internal reflection of light waves (wave-guiding) will occur for incident rays with an angle greater than the critical angle, θ_{crit} (refer Figure 1b). The value of θ_{crit} depends on the refractive indices of the two media. For example if;

n_0 = refractive index of the cured resin ~1.59 at 850nm

n_1 = refractive index of optical fibre

n_2 = refractive index of uncured resin ~1.55 at 850nm

The angle θ_{crit} for the cured/uncured resin interface will be equal to:

$$\sin(\theta_{crit}) = \frac{n_2}{n_0} \quad \text{therefore } \theta_{crit} = 77°.$$

Light rays incident at the cured/uncured resin interface at angles greater than θ_{crit} (Figure 1b.) will be wave-guided through the sensing element by total internal reflection. In order for the sensor to be effective, there must be a total internal reflection at the sensor/composite matrix interface before the light reaches the detector. As the refractive indices of the composite and sensor become equal during the curing process the value of θ_{crit} will approach 90°. At this stage no reflections at the composite matrix/sensor interface are possible and the transmitted light intensity becomes zero.

The gel point of a resin is an important stage during the cure cycle. It marks the point at which the resin has sufficiently reacted causing the viscosity of the resin to become effectively infinite. This means that no further compaction and bleeding of the curing laminate is possible. From the uncured state up until the gel point is perhaps the most important part of the composite processing window. At the gel point the matrix refractive index is 1.57 [12]. At this stage the value of θ_{crit} is equal to about 81°.

It is probably best to make the sensing element short to limit the amount of multiple reflections allowed along it's length. The resin is more likely to create signal loss due to inhomogeneities than the relatively homogeneous optic fibre which has a known rate of loss along it's length.

4.3 Sensor Manufacture and use

The production of such a sensor is perhaps not a trivial exercise. Resin for the cured polymer sensing section could be obtained from the composite prepreg by solvent extraction. The solvent used must be chosen such that it does not interfere with the chemical nature of the resin and that the solvent does not introduce voids within this matrix when it is cured. Voids in the matrix will reduce the intensity of the transmitted light and create false readings. Although any resin will produce some level of voids during cure Afromowitz and Lam found that the cured epoxy is relatively homogeneous. Due to the size of the optic fibre it is envisaged that it may be difficult to bond the matrix segment into place successfully.

Bonding the fibre segment into place is perhaps the most difficult task. The entire arrangement is likely to be quite delicate due to the brittle nature of epoxy resin. A small apparatus needs to be constructed to make the butt-joining of the two optic fibres easier. The sensor (resin) may then cured and removed from the joining apparatus immediately prior to inclusion in the layup.

Difficulties may arise if the sensing section is disturbed during the curing process. This may cause the sensor to break and lose signal.

The sensor and sensing apparatus is likely to comprise of the following elements:

1. Communications grade optical fibre (consumable)
2. Laser diode producing a frequency around 850nm
3. Epoxy resin (consumable)
4. Photodetector
5. Data acquisition system

These items are relatively inexpensive and readily obtained.

5. Conclusion

A review of current cure-monitoring sensors was undertaken and the most promising methods outlined for embedding into composite components. Of all the sensors reviewed the most promising appears to be based on the change in resin refractive index during cure. This type of sensor measures light intensity passing through an optic fibre in which a section of the fibre has been replaced by a segment of cured resin. Light passes through the sensor at the start of the cure but as the refractive index of the composite matrix approaches that of the cured resin the transmission of light decreases until no light passes through the sensor segment signalling that the composite is fully cured. This type of sensing system is inexpensive and is not complex. This technique warrants further investigation into it's applicability as an embedded sensor in composite components.

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19. ABSTRACT

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